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STUDIES TO ESTABLISH KEY PROCESSES PARAMETERS FOR CONVERTING HIGH SULFUR RESIDUE INTO ON-GRADE BITUMEN

KUMAR KAMAL, SINGH ANAND & NEGI SANDEEP

CSIR-Indian Institute of Petroleum, SGRR PG College, Dehradun, India

ABSTRACT

Bitumen of improved performance have better load bearing and spreading ability at high temperatures and are not much stiff so as to crack at low temperatures, besides better adhesion and aging resistance compared to conventional binders. The objective of the study is to make improved quality bitumen through chemical modification/interaction by using air blowing approach using high sulfur feed stocks. The Short residue (SR) of high sulfur used in the study has 5% wt sulfur. For converting high sulfur SR into on grade bitumen, it is subjected to air blowing under varying conditions/processes parameters viz. temperature, duration and air rate. In this paper air blowing was carried out at two different temperatures and two different air rates. Penetration index of different bitumen binders was determined as it is a good parameter to define temperature susceptibility of bitumen. This paper describes how the finished product meets the desired specifications of BIS73-2006.

KEYWORDS: Air Blowing, Asphaltene, bitumen, Conventional Binder, Maltene, Short Residue

INTRODUCTION

Bitumen is a visco-elastic semi solid viscous material and is composed of mainly two components viz. asphaltene and maltene. Asphaltene is dispersed phase and maltene is dispersion medium in a colloidal system of bitumen. The remains of vacuum distillation of crude oil is complex mixture of organic and inorganic compounds. These compounds constitute asphaltenes and maltene parts of bitumen. Asphaltene is n-heptane insoluble part of bitumen and maltene is soluble part of n-heptane[1]. In colloidal system of bitumen the term maltene refers to saturates, naphthene aromatics, polar aromatics and resins[2]. The physico-chemical properties of bitumen depend upon the crude source and composition of base bitumen. Bitumen is temperature susceptible material i.e. the physico-chemical properties of bitumen change with change in temperature. At temperature above the softening point, it acts as a lubricant and at temperature below the softening point, it acts like glue. Below its freezing point it becomes brittle solid[3].

Air-blowing has been used to harden the bitumen and to increase its viscosity as required by a specific grade of bitumen. This approach has however some implications like poor aging resistance and even light air-blowing is employed to modify the base bitumen to make quality grade bitumen.

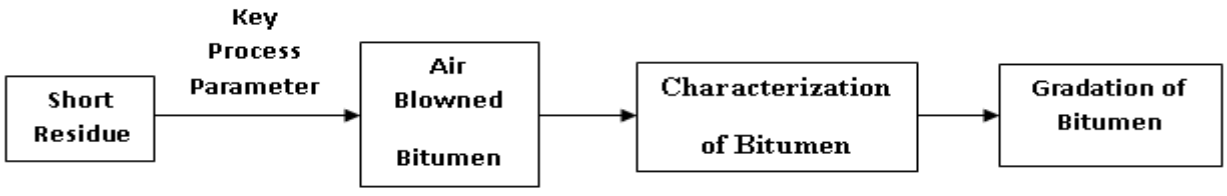

Figure 1: Flow Diagram of Air Blowing of Bitumen

Bitumen is prepared or modified by using different approaches like blending of SR with additives, blending of SR with suitable polymers, blending of base bitumen with fluxes etc. Besides all these air-blowing is one of the most commonly used approaches to modify the properties of base bitumen. But this approach cannot be applied for all base bitumens. For air-blowing base bitumen should have following characteristics:-

- It should have penetration > 150.
- Asphaltene content < 15%
- It should have sufficient aromatics & CCR

Air-blowing involves oxidation, dehydrogenation, polymerization reactions etc. All these reactions result an increase in molecular size of the asphaltene[1]. Air-blowing is carried out under controlled manner of temperature, pressure and duration.

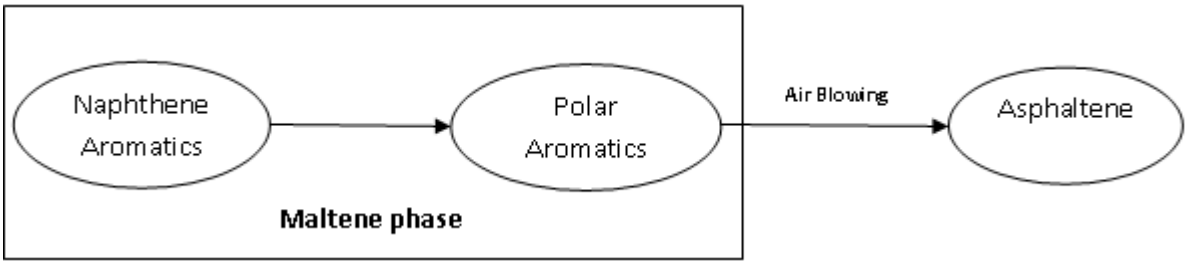

Figure 2: Flow Diagram of Chemical Changes in Bitumen Components During Air Blowing of Base Bitumen

MATERIALS

The short residue (base bitumen) of IOC Barauni refinery of high sulfur was used for this study. The physico-chemical characterization of base bitumen is reported in Table – 1.

Methods

Physico-Chemical Characterization of Base Bitumen

Physico-chemical characterization of base bitumen was carried out as per BIS/ASTM test procedures. The base bitumen was characterized for degree API, penetration, softening point, ductility, and viscosity at 60 °C, kinematic viscosities at 100 and 135 °C, sulfur content and water content etc.

Air-Blowing of Base Bitumen

Melted base bitumen is charged into reaction kettle and the temperature of the kettle increased at the rate of 2 °C/min. When temperature reached to 130 °C, air is blown into the kettle/base bitumen at controlled air-rate for a stipulated period. The temperature of the reaction kettle kept constant at air-blowing temperature for the certain period. Sample is drawn time to time to check its consistency as per required grade of bitumen and then characterized for different tests. The physico-chemical characterization of the air-browned bitumen is mentioned in Table 2 and 3.

RESULT AND DISCUSSIONS

Physico-Chemical Characterization of Base Bitumen

The physico-chemical characterization of base bitumen indicates that the penetration of feed at 25 °C is 200 dmm and ductility at 25 °C is 94.00 cm. High penetration base bitumen indicates that it is suitable to make bitumen via

air-blowing approach. High ductility of base bitumen indicates that the material is aromatic rich. Sulfur content in base bitumen was found to be 4.73 wt%. This indicates that base bitumen has high sulfur content. This indicates that modification of bitumen using air-blowing approach best as this lead to the formation of condensed heterocyclic aromatic compounds during the course of reaction. High value of CCR (21.5) showed that base bitumen is aromatic rich and contained condensed polycyclic aromatic asphaltene type structures. Solubility of base bitumen > 99.00% wt indicated that base bitumen is aromatic rich and has no foreign material which deteriorated the properties of bitumen

Preparation of VG Grade Bitumen from Base Bitumen Via Air-Blowing

Air-blowing of base bitumen is done under varying conditions of temperature (250 & 270 °C), air rate (5 & 7 lit/min/kg) and residence time. The following observations were made from the data reported in Table - 2, 3.

Initially air-blowing was carried out at 250 °C at air-rate of 5.0 lit/min/kg and air blowned samples were taken out at 105 minutes and 210 minutes respectively. The penetration of air-blowned bitumen at 105 and 210 minutes were found 95dmm and 65dmm respectively. The softening point at these durations found 41°C and 43 °C respectively. The value of penetration and softening point of sample taken out at 105 minutes meet the range of VG-10 grade bitumen. The viscosity at 60 °C of sample taken out at 105 minutes and 210 minutes were found 590 and 770.5 poise respectively. Both of the values were quite low (< 800 for VG-10 & < 1600 for VG-20). Thus the two blowned samples taken out were not found in the range of VG-10 & VG-20 grade bitumen.

Table 1: Physico-Chemical Characteristics of Base Bitumen

Characteristics	Base Bitumen
Density, d^{15}_{4}	1.0288
°API	5.95
Penetration at 25 °C	200
Softening Point, °C	39.5
Penetration Index	-0.247
Ductility at 25 °C, cm	100
Viscosity at 60 °C, poise	346.4
Kinematic Viscosity at 100 °C, cSt	1389.5
Kinematic Viscosity at 135 °C, cSt	383.1
Asphaltene, wt %	12.24
Sulfur, wt%	4.73
Flash Point, °C	298
Water content, wt%	0.1
Pour Point, °C	57.0
CCR, wt%	21.5
Solubility in trichloroethylene, wt%	> 99.00

- Similarly another run was given at 270 °C at air rate of 5.0 lit/min/kg and samples were taken out at 120 and 165 minutes respectively. The viscosity of these two samples at 60 °C was found 673.9 and 859.4 poise. These values were quite low and not full fill the specs of neither of the VG grade bitumen.
- When air blowing was carried out at 250 °C at air rate of 7.0 lit/min/kg and sample were taken out at 115 and 180 minutes respectively; penetration, softening point and viscosity at 60 °C found 92 dmm, 44 °C and 1067.6 poises respectively. All these value were found in the range of VG-10 specification of bitumen.

- Penetration, softening point and viscosity at 60 °C of samples were taken out at 180 minutes were found 60 dmm, 46 °C and 1417.8 poise respectively. All these value were found in the range of VG-20 specification of bitumen except viscosity. The viscosity at 60 °C was marginally low i.e. less than 1800 poise.
- When air blowing was carried out at 270 °C at air rate of 7.0 lit/min/kg and sample was taken out at 101 minutes; penetration, softening point and viscosity at 60 °C found 85 dmm, 45 °C and 920.0 poises respectively. Here penetration and softening point were found in the range of VG-10 specification of bitumen.

Table 2: Physico-Chemical Characteristics of Air-Blowned Bitumen at Air Rate of 5.0 L/Min/Kg

Conditions of Air Blowing of Base Bitumen				
Temperature, °C	250 ± 2.0		270 ± 2.0	
Air rate	5.0 lit/min/kg			
Duration, min	105	210	120	165
Characteristics				
Grade	**VG-10**	**VG-20**	**VG-10**	**VG-20**
Penetration at 25°C	95	65	95	75
Softening Point, °C	41	43	41.6	42.5
Penetration Index	-2.291	-2.55	-2.078	-2.380
Viscosity at 60 °C, poise	590	770.5	673.9	859.4
Kinematic Viscosity at 135 °C, cSt	470.8	572.9	411.3	527.7
AFTER RTFOT				
Ductility at 25 °C	100	100	100	100
Viscosity at 60 °C, poise				
Viscosity ratio	4.02	3.55	2.69	2.45

Table 3: Physico-Chemical Characteristics of Air-Blown Bitumen at Air Rate of 7.0 L/Min/Kg

Conditions of Air Blowing of Base Bitumen				
Temperature, °C	250 ± 2.0		270 ± 2.0	
Air rate	7.0 lit/min/kg			
Duration, min	115	180	101	131
Characteristics				
Grade	**VG-10**	**VG-20**	**VG-10**	**VG-20**
Penetration at 25 °C	92	60	85	75
Softening Point, °C	44	46	45	45
Penetration Index	-1.359	-1.84	-1.264	-1.596
Viscosity at 60 °C, poise	1067.6	1417.8	920	1352.9
Kinematic Viscosity at 135 °C, cSt	491	591.1	473.4	525.2
AFTER RTFOT				
Ductility at 25 °C	100	100	100	100
Viscosity at 60 °C, poise	1747.1	3215.3	1045.05	2445.7
Viscosity ratio	1.63	2.26	1.13	1.88

When air blowing was carried out at 270 °C at air rate of 7.0 lit/min/kg and sample were taken out at 131 minutes; penetration, softening point and viscosity at 60 °C found 75 dmm, 45 °C and 1352.9 poises respectively. Here penetration and viscosity at 60 °C were found in the range of VG-20 specification of bitumen.

Penetration Index

Van Doormaal developed an equation for the temperature susceptibility that assumes a value of about zero for road bitumens. Pfeiffer and Van Doormaal found that most bitumens had a penetration of about 800dmm at ASTM

softening point temperature. This is defined by equation 1.

$$\text{Penetration Index} = \frac{1952 - 500 \log Pen - 20\ Softening\ Point}{50 \log Pen - Softening\ Point - 120} \quad (1)$$

Where PI= Penetration Index, SP= Softening Point in ^{0}C, Pen= Penetration in dmm

NOTE

- High PI means low temperature susceptibility
- High PI binder is vulnerable to brittleness, leading to cracks in cold climate areas and prone to rutting at high temperatures.
- Low PI, low PVN and high VTS values are indicators of a binder that is highly temperature susceptible to temperature changes and vice versa.

(Ref; Effect of rheology on the bitumen foamability and mechanical properties of foam bitumen stabilized mixes, By Mofreh F. Saleh, University of Canterbury, dept. of Civil Engineering)

CONCLUSIONS

Based on result and discussion the following conclusions were made:

The prepared VG-10 and VG-20 grade bitumen's meet all specification of conventional bitumen as per IS73:2006, except absolute viscosity at 60 °C in case of VG-20. Physico-chemical characterization of base bitumen showed that base bitumen is suitable for the preparation of VG-10 & VG-20 bitumen as the base bitumen is aromatic rich and has asphaltene type structures compounds. High density, high CCR (21.5 wt %) and asphaltene content indicated that base bitumen is aromatic rich. High sulfur content (4.73 wt %) and high penetration in the range of 200 dmm indicated that air blowing is suitable approach that can be used to prepare VG bitumen. The operating key process parameters temperature 250 °C, 7.0 lit/min/kg of air rate and residence tine of 115 and 180 minutes found suitable to prepare VG-10 and VG-20 grade of bitumen.

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REFERENCES

1. Shell Bitumen Hand Book)\
2. Garcia-Mcrales M, Partal P, Garizalez N, Gonza Uz' O, Mum Oz M.E, Viscous properties of micro structurally of recycled EVA modified bitumen Feul, VI-83, 31-38, 2003
3. Chapter - 2 materials, MTAG Vol-I flexible Pavement preservation 2nd Edition, page 2-1to2-28, Nov-8-2007